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## MEMORANDUM

DATE: 22 December 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle  
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9805598  
Site: Duwamish River

WORK ASSIGNMENT NO. 46-35-0JZZ

WORK ORDER NO 4000-019-038-5200-00

DOC CONTROL NO 4000-019-038-AAAK

cc Bruce Woods, RAP-WAM, U.S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle (memo only)  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of three sediment samples, laboratory batch K9805598, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered.

98344037                      98344043                      98344047

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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Site. Duwamish River

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1 Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria.

2. Detection Limits

Detection limits met project required quantitation limits with the following exceptions.

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98344037	Tetra-n-butyltin	10	20
98344037	n-butyltin	10	20
98344043	Tetra-n-butyltin	10	20
98344043	n-butyltin	10	20

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

3. Initial Calibration

A six-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

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## 5. Blanks

### a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

### b) Field Blanks

No field blanks were associated with this SDG.

## 6. Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, triphenyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	20 - 195%
Triphenyltin	20 - 172%

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following

Sample	Surrogate	Percent Recovery
K980821-DLCS	Triphenyltin	52

Sample results are qualified as estimated (J) when both surrogate recoveries are outside project limits. As only one surrogate was outside QC limits and surrogate recoveries in the original LCS were within QC limits, no qualifiers were assigned to sample results

## 7 Laboratory Control Sample (LCS)

LCS recovery goals for butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for both sediment and porewater Based on conversations with the

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laboratory, historical control chart limits of 20 to 138 percent for water and 20 to 164 percent for sediment were also used for data qualification

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980821-LCS	Di-n-butyltin	40	60-130 (P) 20-164 (L)	98344037 98344043 98344047
K980821-LCS	n-Butyltin	14	60-130 (P) 20-164 (L)	98344037 98344043 98344047
K980821-DLCS	n-Butyltin	4	60-130 (P) 20-164 (L)	98344037 98344043 98344047

The relative percent difference (RPD) value between replicates for Di-n-butyltin was 40 percent and n-Butyltin was 111 percent. Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits. Undetected sample results were qualified as rejected (R) when LCS recoveries were outside both project and laboratory QC limits

#### 8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

No matrix spike/matrix spike duplicate analysis was performed for this SDG. Instead, a replicate laboratory control sample set (LCS/DLCS) was analyzed

#### 9. Field Duplicate Analysis

No field duplicates were associated with this SDG.



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#### 10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. The case narrative noted that the RPD value for the n-butyltin replicate analysis for the LCS/DLCS was outside the QC limits. The variability is attributed to the analytical method, which is known to have poor performance for n-butyltin. The low n-butyltin recoveries in the LCS/DLCS were not discussed in the narrative. No other problems were noted.

#### 11. Laboratory Contact

No laboratory contact was required

#### Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

#### Data Qualifiers

- U - The compound was analyzed for, but was not detected
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification
- N - Presumptive evidence of presence of material (tentative identification).
- I - Elevated reporting limit due to matrix interference

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805598  
**Date Collected:** 8/18/98  
**Date Received:** 8/19/98

Butyltins

A

**Sample Name** 98344043  
**Lab Code** K9805598-001  
**Test Notes** D

**Units** ug/Kg (ppb)  
**Basis** Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	ND	20ug/L
Tri-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	48	
Di-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	22	J
n-Butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	ND	<del>20ug/L</del> R

R-1-

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

10/5/01

1822/020597p

MGT 12/1/98

## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805598  
**Date Collected:** 8/18/98  
**Date Received:** 8/19/98

## Butyltins

Sample Name 98344047 Units ug/Kg (ppb)  
Lab Code K9805598-005 Basis Dry  
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/21/98	8/26/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/21/98	8/26/98	47	
Di-n-butyltin	Method	Butyltins	5	5	8/21/98	8/26/98	8 J	
n-Butyltin	Method	Butyltins	5	5	8/21/98	8/26/98	18 <del>R</del> J	Run

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

10/6/98

1S22/020597p

10/12/98  
00067

**COLUMBIA ANALYTICAL SERVICES, INC.**

**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805598  
**Date Collected:** 8/18/98  
**Date Received:** 8/19/98

**Butyltins**

**Sample Name** 98344037 **Units** ug/Kg (ppb)  
**Lab Code** K9805598-009 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	ND	20 ug/L
Tri-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	180	
Di-n-butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	44	J
n-Butyltin	Method	Butyltins	20	20	8/21/98	8/25/98	ND	20 ug/L J R

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

10/5/98

1572/020597p

NOT 12/8/98

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